



## Zero *trans* fats from soybean oil and fully hydrogenated soybean oil: Physico-chemical properties and food applications

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### ABSTRACT

Blends of soybean oil (SO) and fully hydrogenated soybean oil (FHSBO), with 10%, 20%, 30%, 40% and 50% FHSBO (w/w) content were interesterified under the following conditions: 0.4% sodium methoxide, 500 rpm stirring, 100 °C, 20 min. The original and interesterified blends were examined for triacylglycerol composition, melting point, solid fat content (SFC) and consistency. Interesterification caused considerable rearrangement of triacylglycerol species, reduction of trisaturated triacylglycerol content and increase in monounsaturated and diunsaturated triacylglycerols, resulting in lowering of respective melting points. The interesterified blends displayed reduced SFC at all temperatures and more linear melting profiles as compared with the original blends. Yield values showed increased plasticity in the blends after the reaction. Isosolid diagrams before and after the reaction showed no eutectic interactions. The 90:10, 80:20, 70:30 and 60:40 interesterified SO:FHSBO blends displayed characteristics suited to application, respectively, as liquid shortening, table margarine, baking/confectionery fat and all-purpose shortenings/biscuit-filling base.

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### 1. Introduction

Most natural oils and fats offer limited application in their unaltered state, due to their particular fatty acid and triacylglycerol composition (Chiu, Gioielli, & Grimaldi, 2008; Rozendall, 1992). Accordingly, oils and fats are modified chemically by hydrogenation or interesterification; or physically, by fractionation (Erickson, 1995; Fattahi-far, Sahari, & Barzegar, 2006).

Although used for a long time, partial hydrogenation also results in substantial formation of *trans* fatty acids, compounds that act as coronary artery disease risk factors by modulating the synthesis of cholesterol and its fractions and acting on the eicosanoids. A number of studies have suggested a direct relationship between *trans* isomers and increased risk of vascular disease. In response, many health organizations have recommended reducing consumption of foods containing *trans* fatty acids (Enig, 1996; Gurr, 1990; Hunter, 2005; Lichtenstein, 1993; Mensink & Katan, 1990).

In this connection, chemical interesterification has proved the main alternative for obtaining plastic fats that have low *trans* isomer content or are even *trans* isomer free. Unlike partial hydrogenation, this process does not isomerize the fatty acids' double bonds and does not affect their degree of saturation (Haummann, 1994; Norizzah, Chong, Cheow, & Zaliha, 2004; Ribeiro, Moura, Grimaldi, & Gonçalves, 2007). In the interesterification, the fatty

acids are not altered, but are redistributed in the triacylglycerol molecules. The process thus consists in simultaneously breaking the existing ester bonds and forming new bonds in the glycerol molecules (Rozendall, 1992). In this way, chemical interesterification modifies the triacylglycerol composition of the oil or fat and thus its physical properties, crystallization and melting behavior, solid fat content (SFC), texture and crystal habit, contributing to greater availability of oil fractions for food product applications (Dian, Sundram, & Idris, 2007).

A number of studies have investigated the influence of chemical interesterification on the physico-chemical properties of oils and fats or their blends, including the work of Gioielli and Baruffaldi (1988), List, Emken, Kwolek, Simpson, and Dutton (1977), List, Mounts, Orthoefer, and Neff (1995), Lida and Ali (1998), Marangoni and Rousseau (1998a, 1998b), Petrauskaite, De Greyt, Kellens, and Huyghebaert (1998), Kok, Fehr, Hammond, and White (1999), Khattoon (2000), Rodríguez, Castro, Salinas, López, and Miranda (2001), Rodrigues, Gioielli, and Anton (2003), Rodrigues and Gioielli (2003), Norizzah et al. (2004), Karabulut, Turan, and Ergin (2004), Ramli, Said, and Loon (2005), Grimaldi, Gonçalves, and Ando (2005), Khattoon and Reddy (2005), Silva and Gioielli (2006), Piska, Zárubová, Louzecký, Karami, and Filip (2006), and others. The interesterification of liquid oils with hardfats is the most versatile way for producing zero *trans* fats, yielding fat bases with excellent characteristics for preparation of margarines, shortenings and spreads (Karabulut et al., 2004; Khattoon & Reddy, 2005; List et al., 1995; Lo & Handel, 1983).

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In world vegetable oil consumption, soybean oil (SO) stands out for its nutritional qualities, permanent supply, considerable economic value and high functionality, making it a particularly interesting raw material for producing special fats (O'Brien, 2004). Fully hydrogenated soybean oil (FHSBO), a relatively low-cost product of total hydrogenation, can be used as *hardstock* for producing interesterified fat bases. Zeitoun, Neff, List, and Mounts (1993) report on a chemical interesterification of blends formulated with FHSBO and nine different vegetable oils, at a ratio of 1:1 (w/w).

In addition to its economic feasibility, the advantage of using FHSBO as *hardstock* derives from its high (approximately 85%) content of stearic acid, which is not atherogenic and therefore has no adverse effect on cardiovascular disease risk (Hunter, 2005; O'Brien, 2004; Rao & Lokesh, 2003). Although a saturated fatty acid, consumption of stearic acid does not significantly alter total serum cholesterol and LDL cholesterol levels, unlike most – lauric, myristic and palmitic – saturated fatty acids. Numerous clinical studies have shown that the effect of stearic acid on plasma lipoprotein concentrations is similar to that of oleic acid (Dirienzo, Lemke, Petersen, & Smith, 2008; Mensink, 2005; Nielsen, 2006; Sampath & Ntambi, 2005; Sanders & Berry, 2005; Tholstrup, 2005). Therefore, FHSBO is used in producing interesterified fats with a view to the stability and functionality required of fats for food applications and does not entail adverse health effects.

Even though chemical interesterification is extremely functional from the technological point of view, replacing partially hydrogenated fats in food products, particularly in shortenings and confectionery products, does pose challenges, because suitable SFC curves, plasticity, crystallization properties and texture are difficult to obtain in the absence of *trans* fatty acids. Accordingly, the study of, and application guidelines for, interesterified fats must comprehend jointly their fundamental physico-chemical properties, as regards triacylglycerol composition, SFC, consistency and melting point (O'Brien, 2004; Reyes-Hernandez, Dibildox-Alvarado, Charo-Alonso, & Toro-Vazquez, 2007).

The purpose of this study was to evaluate the chemical interesterification of binary blends of SO:FHSBO, with 10%, 20%, 30%, 40% and 50% FHSBO content, with a view to studying fat bases for application in food products. Results for triacylglycerol composition, melting point, SFC, isosolid diagram and consistency were analyzed, before and after randomization.

## 2. Materials and methods

### 2.1. Raw materials

The materials used were refined soybean oil (SO), purchased in a local store, and fully hydrogenated soybean oil (FHSBO), kindly provided by a regional supplier. The catalyst was 99%-pure sodium methoxide powder (Sigma-Aldrich).

### 2.2. Blends

The blends were prepared in the proportions 90:10, 80:20, 70:30, 60:40 and 50:50 SO:FHSBO (w/w), melted at 100 °C and homogenized for 10 min at that temperature to melt the crystals completely, prior to each interesterification reaction.

### 2.3. Chemical interesterification

For the reactions, a 500 mL, jacketed, borosilicate glass reactor with bottom drain port and conical ground glass joints was coupled to a thermostated circulator bath (LAUDA RE 212, –30 to +200 °C, +0.02 °C), stirring system (universal motor with electronic speed control up to 4000 rpm – Marconi, BR) with axial flow stir-

rer, vacuum pump (Vacuubrand model 30 diaphragm pump) and probe-type digital thermometer (–50 to +300 °C, ±1 °C – Inco-term). The samples (200 g) were dried for 20 min at 100 °C in the reactor itself, under vacuum with stirring at 500 rpm. Catalyst content was 0.4% and the reaction was conducted under vacuum at 100 °C, with stirring at 500 rpm, for 20 min, according to the optimization by Grimaldi et al. (2005). The reaction was terminated by adding distilled water and 5% citric acid solution. The interesterified samples were washed carefully with distilled water (80 °C) to remove any soaps that had formed and were then dried under vacuum at 110 °C for 30 min.

### 2.4. Fatty acid composition

Analysis of fatty acid composition was performed in a capillary gas chromatograph (CGC Agilent 6850 Series GC System) after esterification using the method of Hartman and Lago (1973). The fatty acid methyl esters were separated according to AOCS procedure 2-66 (AOCS, 2004) in a DB – 23 Agilent capillary column (50% cyanopropyl-methylpolysiloxane), dimensions 60 m, Ø int: 0.25 mm, 0.25 µm film. Oven temperature was 110 °C–5 min, 110–215 °C (5 °C/min), 215 °C–24 min; detector temperature: 280 °C; injector temperature 250 °C; carrier gas:helium; split ratio 1:50; injection volume: 1.0 µL. Qualitative composition was determined by comparing peak retention times with the respective standards for fatty acids.

### 2.5. Iodine value

It was calculated from the fatty acid composition by AOCS Method Cd 1c-85 (AOCS, 2004).

### 2.6. Triacylglycerol composition

Triacylglycerol composition was analyzed in a CGC Agilent 6850 Series GC System capillary gas chromatograph. A DB-17HT Agilent Catalog: 122-1811 capillary column (50%-Phenyl-methylpolysiloxane, 15 m in length × 0.25 mm bore and containing 0.15 µm film). The conditions were: split injection, ratio 1:100; column temperature: 250 °C, programmed up to 350 °C at 5 °C/min; carrier gas: helium, at 1.0 mL/min flow rate; injector temperature: 360 °C; detector temperature: 375 °C; injection volume: 1.0 µL; sample concentration: 100 mg/5 mL of tetrahydrofurane. Triacylglycerol groups were identified by comparing retention times, following the procedures of Antoniosi Filho, Mendes, and Lanças (1995).

### 2.7. SFC

This was determined using Nuclear Magnetic Resonance (NMR) spectrometry (Bruker pc120 Minispec) and TCON 2000 high precision dry baths (0–70 °C) (Duratech, USA). AOCS Procedure Cd 16b-93: direct method, taking readings from samples in series at temperatures of 10, 20, 25; 30, 35, 40, 45, 50, 55 and 60 °C, with tempering for non-stabilized fats (AOCS, 2004). In addition, it was necessary to modify the standard tempering prescribed by AOCS Method Cd 16b-93 in order to ensure stabilization of the crystallization of the interesterified blends: the samples were kept at 0 °C for 2 h and at each reading temperature for 1 h.

### 2.8. Construction of isosolid diagrams

The isosolid line diagrams (i.e. showing compositions in which the SFC values of a blend are equivalent at a given temperature) were constructed from the data supplied experimentally by NMR using the modified tempering method (Braipson-Danthine & Deroanne, 2006).

## 2.9. Melting point

This was determined using an open capillary tube, to AOCS standards, Method Cc 3–25 (AOCS, 2004). In addition, melting point was calculated for the temperature corresponding to 4% solids content, obtained from the SFC curve given by NMR using the modified tempering described above, by way of polynomial equations adjusted with the aid of Statistica 6.0 software (Karabulut et al., 2004; Statistica, 2002). A simple linear regression model was applied to relate the data obtained by the two methods.

## 2.10. Consistency

Consistency was determined with a PC-controlled TA-XT2i texture analyzer (Stable Micro Systems) using an acrylic cone with an apex angle of 40° and no truncation. The samples were heated in a microwave oven to approximately 80 °C to completely melt the crystals and placed in 50 mL beakers. Conditioning was performed in an incubator for 24 h at 5 °C to crystallize the fats and then for 24 h at the read temperatures: 5, 10, 15, 20, 25, 30, 35, 40 °C or higher, according to the sample analyzed (Rodrigues et al., 2003). The tests were conducted under the following conditions: distance = 10 mm; speed = 2 mm/s; time = 5 s; and five readings for each sample. From these conditions the compression force was obtained in (gf). The penetration data were converted into yield values, following Haighton (1959).

## 3. Results and discussion

### 3.1. Fatty acid composition in raw materials and blends

Table 1 shows the fatty acid composition and iodine value calculated for the raw materials and blends used in the chemical interesterification. These results express the mean of the two assays and agree with the limits encountered in the literature (Erickson, 1995; O'Brien, 2004). For the SO, the predominant fatty acids were linoleic acid (54.87%), oleic acid (23.17%) and palmitic acid (11.37%). The FHSBO was high in stearic acid content (86.62%), followed by palmitic acid (11.50%). This value lies between the 83.70% and 90% reported by List et al. (1995) and Lee, Akoh, and Lee (2008) for this raw material.

The percentages of unsaturated fatty acids in the SO and FHSBO were 84.02% and 0.29%, respectively and, in the blends, were between 75.68% and 41.44%. Adding FHSBO to the SO produced blends with stearic acid contents between 11.80% and 45.79%.

The raw materials and, therefore, the blends, were free of *trans* fatty acids, which is of considerable importance from the nutritional point of view. The iodine values calculated for the SO and FHSBO were within the ranges of the literature and, for the blends, are proportional to the amounts of each component (Erickson, 1995; Gunstone & Harwood, 2007).

The chemical interesterification process neither affected the degree of saturation nor caused isomerization of the fatty acid double bonds. The fatty acid composition of the starting materials was thus not altered (Karabulut et al., 2004; Lida, Sundram, Siew, Aminah, & Mamot, 2002; Rozendall, 1992).

### 3.2. Triacylglycerol composition

Analysis of triacylglycerol composition represents a true indication of randomization, and is extremely useful for monitoring modification of interesterified fats and outlining specific applications for them (O'Brien, 2004). Table 2 shows the main individual triacylglycerols that make up the SO and FHSBO raw materials and the blends, before and after chemical interesterification. The initials represent the *sn*-1, *sn*-2 and *sn*-3 positions of the triacylglycerols. For the SO, 17 triacylglycerol species were found and, for the FHSBO, only 4. The LLL, OLL and PLL triacylglycerols predominated in the SO, accounting for 55.67% of total triacylglycerol composition. These results agree with those presented by Gunstone and Harwood (2007) for the triacylglycerol composition of SO. For the FHSBO, only trisaturated triacylglycerols were found, with StStSt predominating (63.35%), followed by PStSt, with 30.79% of total triacylglycerols present. Humphrey and Narine (2004) found very similar contents for this fraction, with these two preponderant triacylglycerols totaling 90%. LLL and OLL were the predominant triacylglycerols in the 90:10 SO:FHSBO blend, while LLL and StStSt were the triacylglycerol species with highest percentages in the 80:20 and 70:30 SO:FHSBO blends. The saturated triacylglycerols PStSt and StStSt were the predominant species in the 60:40 and 50:50 SO:FHSBO blends.

Interesterification produced significant alteration in the triacylglycerol composition of the blends studied. Randomization resulted in the formation of new triacylglycerols, such as StOSt, and considerable StLSt contents (as a result of the high concentrations of oleic and stearic acids present in the starting blends), as well as substantial increases in PLSt, StLO and StLL triacylglycerol species, in all the blends evaluated. Generally, interesterification resulted in the formation and increased content of lower melting point, mixed triacylglycerols, as in the results obtained by Norizzah

**Table 1**  
Fatty acid composition (%) and iodine value of the raw materials (SO and FHSBO) and blends.

Fatty acid (%)	SO	SO:FHSBO (%w/w)					FHSBO
		90:10	80:20	70:30	60:40	50:50	
C14:0 – Myristic acid	0.08	0.09	0.09	0.10	0.10	0.10	0.12
C16:0 – Palmitic acid	11.37	11.37	11.38	11.39	11.31	11.38	11.50
C16:1 – Palmitoleic acid	0.08	0.08	0.07	0.06	–	–	–
C18:0 – Stearic acid	3.46	11.80	20.39	28.82	37.54	45.79	86.62
C18:1 – Oleic acid	23.17	20.59	18.41	16.03	13.77	11.48	0.11
C18:2 – Linoleic acid	54.87	49.59	44.08	38.55	32.69	27.33	0.18
C18:3 – Linolenic acid	5.32	4.82	4.27	3.75	3.16	2.65	–
C20:0 – Arachidic acid	0.38	0.40	0.44	0.48	0.54	0.56	0.74
C20:1 – Gadoleic acid	0.24	0.21	0.19	0.17	0.14	0.11	–
C22:0 – Behenic acid	0.51	0.49	0.50	0.48	0.54	0.53	0.53
C24:0 – Lignoceric acid	0.18	0.17	0.17	0.18	0.20	0.20	0.19
Σ Saturates	15.98	24.32	32.97	41.45	50.23	58.56	99.71
Σ Unsaturates	84.02	75.68	67.03	58.55	49.77	41.44	0.29
IV*	135	122	108	95	80	67	0.4

IV\* = Iodine value (g I<sub>2</sub>/100 g).

**Table 2**  
Triacylglycerol composition\* (%) of the SO, FHSBO and blends, before and after interesterification.

TAG (%)	SO	SO:FHSBO (%w/w)										FHSBO
		90:10	90:10-I**	80:20	80:20-I	70:30	70:30-I	60:40	60:40-I	50:50	50:50-I	
PPSt	–	0.48	0.60	0.87	0.81	1.23	1.47	1.86	2.03	2.25	2.63	4.17
POP	0.81	0.73	1.00	0.59	0.79	0.46	0.87	0.46	0.90	0.36	0.62	–
PLP	3.02	2.40	2.31	2.09	2.09	1.74	2.07	1.48	1.90	1.25	1.51	–
PStSt	–	3.93	–	6.84	1.39	10.20	2.42	13.07	5.37	16.00	10.40	30.79
POSt	0.22	–	<b>2.53</b>	–	<b>2.84</b>	–	<b>4.43</b>	–	<b>4.46</b>	–	<b>3.58</b>	–
POO	3.55	3.23	–	2.72	–	2.38	–	1.92	–	1.60	–	–
PLSt	0.67	<b>0.62</b>	<b>6.09</b>	<b>0.52</b>	<b>8.33</b>	<b>0.45</b>	<b>9.67</b>	<b>0.37</b>	<b>10.36</b>	<b>0.30</b>	<b>9.29</b>	–
PLO	10.38	8.92	8.14	7.67	6.34	6.94	4.63	5.80	3.41	4.66	3.12	–
PLL	15.62	13.19	10.56	11.74	8.78	10.07	6.75	8.07	5.02	6.83	4.52	–
PLnL	2.82	2.55	2.88	2.16	1.80	1.76	0.53	1.85	0.74	1.26	–	–
StStSt	–	<b>9.36</b>	–	<b>15.39</b>	<b>1.45</b>	<b>22.37</b>	<b>2.37</b>	<b>28.75</b>	<b>4.18</b>	<b>35.46</b>	<b>11.47</b>	63.35
StOSt	–	–	<b>1.41</b>	–	<b>2.60</b>	–	<b>3.47</b>	–	<b>7.50</b>	–	<b>8.54</b>	–
SOO	0.76	0.48	–	0.69	–	–	–	–	–	–	–	–
StLSt	–	–	<b>1.34</b>	–	<b>7.52</b>	–	<b>11.71</b>	–	<b>15.32</b>	–	<b>15.48</b>	–
OOO	3.79	<b>3.17</b>	–	<b>3.26</b>	–	<b>2.46</b>	–	<b>2.21</b>	–	<b>1.59</b>	–	–
StLO	1.68	<b>1.42</b>	<b>8.70</b>	<b>1.47</b>	<b>10.81</b>	<b>1.10</b>	<b>14.17</b>	<b>0.99</b>	<b>11.44</b>	<b>0.71</b>	<b>7.30</b>	–
OLO	10.25	8.53	3.35	8.05	2.07	7.07	1.79	5.77	1.37	4.46	0.98	–
StLL	1.09	<b>0.95</b>	<b>14.39</b>	<b>0.89</b>	<b>15.09</b>	<b>0.79</b>	<b>14.06</b>	<b>0.64</b>	<b>13.13</b>	<b>0.50</b>	<b>10.28</b>	–
OLL	18.99	17.26	15.75	14.38	11.16	12.29	7.61	11.22	5.41	9.58	4.12	–
LLL	21.06	18.43	16.30	16.68	12.92	15.02	9.67	12.27	6.39	10.69	5.17	–
LnL	5.02	4.37	4.63	3.97	3.19	3.65	2.30	3.26	1.05	2.49	1.00	–
StStA	–	–	–	–	–	–	–	–	–	–	–	1.69
ALL	0.25	–	–	–	–	–	–	–	–	–	–	–

P = palmitic acid; St = stearic acid; O = oleic acid; L = linoleic acid; Ln = linolenic acid; A = arachidic acid.

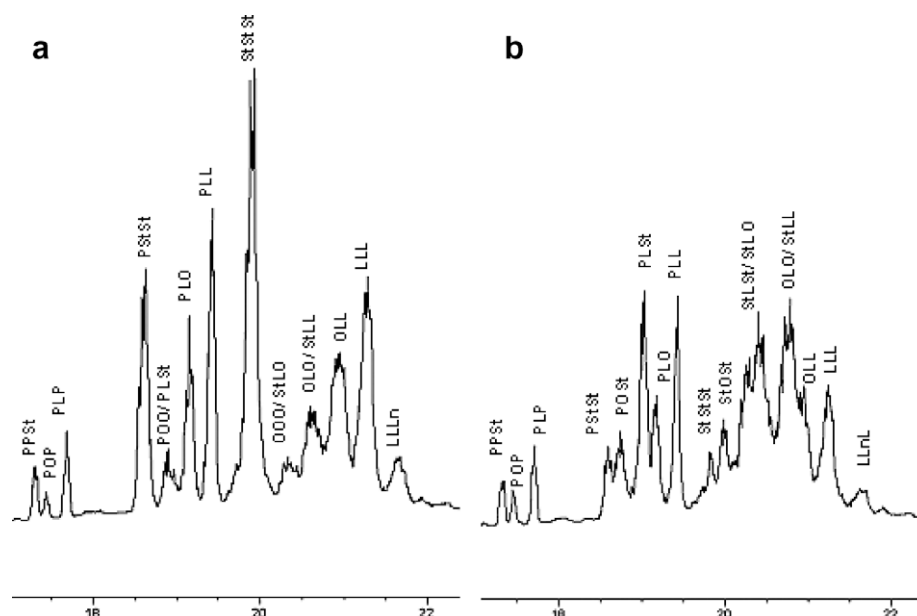
I\*\*: Interesterified blend; –: not detected; \*average value between duplicate samples injection.

et al. (2004) in the interesterification of palm stearin and palm kernel olein blends. Fig. 1 shows the chromatographic profile of the triacylglycerol composition of the 70:30 SO:FHSBO blend before and after chemical interesterification.

In particular, the percentage increase in StLO and StLL triacylglycerols and the formation of significant amounts of StLSt and StOSt species endow the interesterified blends with properties of lubricity, aeration and gloss; while the presence of PPSt and PstSt triacylglycerols, even in small proportions, ensures important qualities as regards structure and moisture barrier, contributing to the functionality of the randomized fatty bases (Ghotra, Dyal, & Narine, 2002; O'Brien, 2004). POO and OOO triacylglycerols, on

the contrary, were not detected after interesterification in any of the blends. Also, StStSt content declined substantially as a result of randomization, to percentages of 1.45%, 2.37%, 4.18% and 11.47%, respectively, in the interesterified 80:20, 70:30, 60:40 and 50:50 SO:FHSBO blends, corresponding to reductions ranging from 90.58% to 67.65%. StStSt was not detected in the interesterified 90:10 SO:FHSBO blend. Fats with low StStSt content are desirable for food applications, because this triacylglycerol is associated with a disagreeable waxy mouthfeel (O'Brien, 2004).

For food product formulation, the physical properties of a fat are more easily interpreted when triacylglycerols are designated by their degree of unsaturation: S<sub>3</sub> (trisaturates), S<sub>2</sub>U (disaturates–



**Fig. 1.** Chromatographic profile of the triacylglycerol composition of the 70:30 SO:FHSO blend before (a) and after (b) interesterification. P = palmitic acid; St = stearic acid; O = oleic acid; L = linoleic acid; Ln = linolenic acid.



**Table 3**

Classes of triacylglycerols in the SO:FHSBO blends, before and after interesterification.

TAG (%)	SO:FHSBO (%w/w)									
	90:10	90:10-I <sup>†</sup>	80:20	80:20-I	70:30	70:30-I	60:40	60:40-I	50:50	50:50-I
S <sub>3</sub>	13.77	0.60	23.36	3.65	34.32	6.26	44.07	11.58	54.11	24.50
S <sub>2</sub> U	3.74	14.68	3.20	24.17	2.65	32.63	2.31	40.45	1.91	39.02
U <sub>2</sub> S	31.08	45.39	27.35	43.40	23.05	40.14	19.27	33.75	15.56	25.22
U <sub>3</sub>	51.41	39.33	46.09	28.78	39.98	20.96	34.35	14.22	28.42	11.26

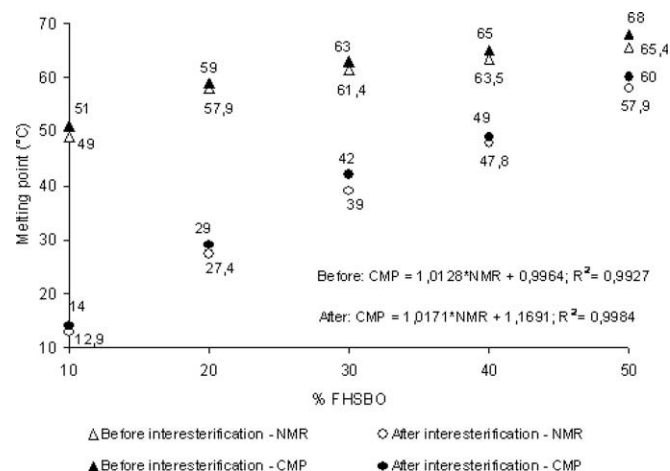
<sup>†</sup>: Interesterified blend.

monounsaturates), U<sub>2</sub>S (monosaturates–diunsaturates) and U<sub>3</sub> (triunsaturates), instead of by the individual triacylglycerol species (Neff, Byrdwell, & List, 2001; Petrauskaite et al., 1998). Table 3 shows the triacylglycerol composition of the blends by S<sub>3</sub>, S<sub>2</sub>U, U<sub>2</sub>S and U<sub>3</sub> content, before and after interesterification. The higher concentration of FHSBO in the blends is associated with higher S<sub>3</sub> content and progressive decline in the other classes of triacylglycerols. In all the blends, interesterification produced significant decreases in S<sub>3</sub> triacylglycerol content and, to a lesser extent, in U<sub>3</sub>-type triacylglycerols, with a concomitant increase in the percentages of mainly S<sub>2</sub>U and U<sub>2</sub>S triacylglycerols. Randomization caused the largest variations in S<sub>3</sub> and S<sub>2</sub>U triacylglycerol classes for all the blends, which corresponded mainly to joint reductions in StStSt/PStSt triacylglycerols and the formation of StLSt/StOSt triacylglycerols. Khatoon and Reddy (2005) encountered a similar effect on triacylglycerol composition in the interesterification of palm stearin with fats from the species *Magnifera indica* and *Madhuca latifolia*, known as Mango fat and Mowrah butter, respectively (Lipp & Anklam, 1998). List et al. (1995) studied the interesterification of an 80:20 SO:FHSBO blend, with a view to production of margarines and shortenings. S<sub>3</sub>, S<sub>2</sub>U, U<sub>2</sub>S and U<sub>3</sub> contents before the reaction were 23.3%, 2.5%, 27.4% and 45.3%, respectively. After randomization, the percentages of these triacylglycerol classes were, respectively, 3.3%, 20.4%, 46.9% and 28.9%, which are very close to those found in this study.

According to Rodrigues and Gioielli (2003), the properties of fatty foods can be related to the triacylglycerol composition of the fat they contain. The S<sub>3</sub> triacylglycerols, with melting points from 54 to 65 °C, and some S<sub>2</sub>U triacylglycerols, with melting points from 27 to 42 °C, are responsible for these products' structure. The U<sub>2</sub>S triacylglycerols are important to their mouthfeel and their functionality at room temperature. The U<sub>3</sub> triacylglycerols, with melting points between –14 and 1 °C, contribute to food softness (Bessler & Orthoefer, 1983; Wiedermann, 1978). Thus, the increased S<sub>2</sub>U and U<sub>2</sub>S content in the SO:FHSBO blends produced by chemical interesterification is associated with enhanced technological functionality, improved sensorial properties and, therefore, greater potential for applying these interesterified bases in foods.

### 3.3. Melting point

Melting point is a parameter of significant importance for characterizing and developing interesterified fats (Nichols & Sanderson, 2003). Timms (1985) and Karabulut et al. (2004) report that fat moves in the capillary tube when there is approximately 4–5% of solid fat, making it possible to determine the melting point at which the SFC is in this range. Fig. 2 shows the melting point values (°C), by FHSBO content of the starting and interesterified blends, determined by the open capillary tube method and calculated from the SFC curve obtained by NMR, as well as the mathematical correlations between the methods. The melting point values determined by SFC curves are lower than those given by the capillary tube method, but correlation between the methods was good, with coefficients of determination ( $R^2$ ) greater than 0.99. This corroborates



**Fig. 2.** Melting points (°C) of the SO:FHSBO blends before and after chemical interesterification, by FHSBO concentration (%). NMR: melting point (°C) calculated from the SFC curve (Karabulut et al., 2004); CMP: melting point obtained by AOCS Method Cc 3-25 (AOCS, 2004).

Nuclear Magnetic Resonance as an appropriate method for determining melting points of fats, because it differs in being quick and easy, and can be used to obtain practically instant measurements. In this study, the results will be discussed in terms of the melting points calculated from the SFC curves.

Adding FHSBO to SO raised the melting point of the blends to values ranging from 49.0 °C to 65.4 °C. The greatest increase in melting point among the samples was seen in the 90:10 and 80:20 SO:FHSBO blends.

Intesterification lowered the melting point of all the blends evaluated, by reducing the proportion of high melting point, trisaturated triacylglycerol; and increasing the percentages of disaturated–monounsaturated and monosaturated–diunsaturated triacylglycerols, which have intermediate melting points (Norizzah et al., 2004; Wiedermann, 1978). Other researchers have reported similar results (Lo & Handel, 1983; Petrauskaite et al., 1998; Rodríguez et al., 2001). Rousseau and Marangoni (2002) found a directly proportional relationship between trisaturated triacylglycerol content and melting point. When interesterification is associated with a reduction in the percentages of triacylglycerol species such as StStSt and PStSt, with melting points of 65 °C and 61.1 °C, respectively, the decrease in melting point is considerable.

The absolute variation in melting points of the interesterified blends in relation to the original blends decreased with FHSBO concentration, to between 36.0 °C and 7.5 °C. It was observed that the greater the ratio between the percentages of S<sub>3</sub> and U<sub>3</sub>-type triacylglycerols in the blends, the smaller was the effect of interesterification on the change in melting point. This behavior was also observed by Rousseau and Marangoni (1998) in relation to milk fat concentration in blends interesterified with canola oil. According to Rozendall (1992) and Dian et al. (2007) this effect is associated with randomization of highly saturated raw materials (such as fully hydrogenated fats) with liquid oils.

The interesterified blends displayed a wide range of melting points (12.9–57.9 °C). Fats with melting points lower than body temperature can be applied directly as shortenings, because they melt completely in the mouth and produce no waxy sensation during consumption (Ghotra et al., 2002; Karabulut et al., 2004). Thus the interesterified 90:10 and 80:20 SO:FHSBO blends, with melting points of 12.9 and 27.4 °C, fall within this group. In addition, the 90:10 SO:FHSBO blend displays the characteristics of liquid shortenings, which can be readily pumped and bottled at low temperatures and have melting points between 10 °C and 15 °C (O'Brien, 2004). The interesterified 70:30 SO:FHSBO blend, whose melting point is 39 °C, lies within the range of most fatty bases for producing solid and semi-solid shortenings, generally represented by the all-purpose shortenings, used mainly in confectionery and bakery products and whose representative melting point is 42 °C. The melting points of the interesterified blends agree with those found by Grimaldi, Gonçalves, and Esteves (2000) in characterizing Brazilian commercial fats for various different industrial purposes, where the great majority of the samples evaluated had melting points ranging from 21.9 °C to 49.2 °C. A recent study by Karabulut and Turan (2006) found that the melting points of 25 samples of margarines and shortenings ranged from 31.2 °C to 43.0 °C. However, the interesterified 50:50 SO:FHSBO blend was found to have too high a melting point (57.9 °C) for food applications. It would thus seem to be restricted to use as a source of zero *trans* hardstock (Lampert, 2000; List et al., 1995).

### 3.4. SFC

SFC curves give good indications of a fat's overall behavior and are useful in formulating and developing new products. The solids content is responsible for many characteristics of margarines and shortenings, including their appearance, ease of packaging, spreadability, oiling-out and organoleptic properties (Lida et al., 2002).

Fig. 3 shows SFC curves for the original and interesterified blends obtained using the standard tempering of AOCS Method Cd 16b-93 (AOCS, 2004) and with the following sample tempering history: 2 h at 0 °C and 1 h at each read temperature. The tempering prescribed by the official method proved suitable for the starting blends, but was not satisfactory for achieving stable crystallization of the interesterified blends, resulting in SFC curves with atypical behavior for the interesterified blends, as can be seen in Fig. 3b. This effect can be seen mainly in the depression in the SFC curve between 20 and 25 °C. This behavior has been encountered in our laboratory in a number of SO:FHSBO blend-based interesterified samples, with FHSBO content higher than 20%, interesterified under a variety of reaction parameters. There is no information on this kind of differentiated behavior in the relevant literature. However, the interesterified blends may be regarded as requiring additional tempering time in order to complete polymorphic stabilization, mainly as a result of their triacylglycerol composition, given that the high content of StLSt, StLO and StLL type triacylglycerols, produced by randomization, seems to be associated with the samples' incomplete crystallization stabilization in the time intervals set by AOCS Method Cd 16b-93 (AOCS, 2004), as in the case of certain special fats and cocoa butter, which require differential tempering because of high StOSt content (Campos, 2005).

According to Dijkstra, Christie, and Knothe (2007), small variations in the temperatures and times used in tempering can result in significant variations in SFC values. Therefore, the tempering conditions were modified accordingly to ensure stable crystallization of the interesterified samples in order to construct the SFC curves. These results were used in the interpretation and discussion of results below.

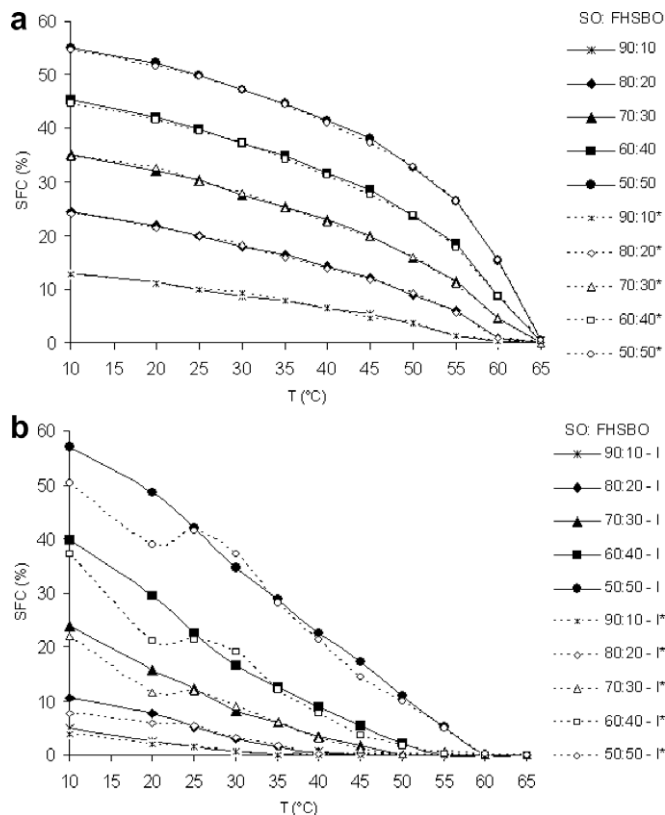


Fig. 3. SFC curves of the SO:FHSBO blends, before (a) and after (b) chemical interesterification, by standard tempering (°) of AOCS Method Cd 16b-93 (AOCS, 2004) and modified tempering (2 h at 0 °C and 1 h at each read temperature).

The original samples gave identical SFC curves for the two tempering methods used. The results of the SFC curves show that the blends have different profiles, covering varying ranges of SFC by temperature. The SFC of the blends was directly proportional to the addition of FHSBO to SO, at all the temperatures considered. At 10 °C, the blends showed SFC ranging from 12.89% to 55.09%, which decreased non-linearly until melting completely at between 60 °C and 65 °C. The greatest decrease in SFC occurred from 50 °C onwards, due to the large proportion of trisaturated triacylglycerols that melt and solubilize at relatively high temperatures.

Interesterification resulted in decreased SFC in the blends at all temperatures. At 35 °C, the SFC of the blends ranged from 7.75% to 44.70%; after randomization, the corresponding values were 0.28–28.72%. This effect is associated with a reduction in trisaturated triacylglycerols (PStSt and StStSt) and a simultaneous increase in percentages of disaturated–monounsaturated and monosaturated–diunsaturated triacylglycerols, represented mainly by StOSt/StLSt and StLO/StLL species, respectively (Fattahi-far et al., 2006; Rousseau, Forestiere, Hill, & Marangoni, 1996; Rousseau & Marangoni, 1998). Due to the formation of mixed triacylglycerols with intermediate melting point, the interesterified blends showed a marked decline in SFC between 25 °C and 45 °C, and the format of the curves was significantly modified by interesterification, which generated more linear melting profiles with more pronounced slope, similar to results obtained by Dian et al. (2007) in interesterification of palm stearin/sunflower oil/palm kernel olein. According to Rousseau and Marangoni (1998), the linearization of SFC curves after randomization is a consequence of the greater variability of the triacylglycerol species resulting from the reaction.

The largest alterations in SFC of the interesterified blends as compared with the original blends were observed at 20, 30, 40, 45 and 50 °C for the 90:10, 80:20, 70:30, 60:40 and 50:50 SO:FHS-

BO blends, respectively, as a result of the high proportion of triacylglycerols in the randomized blends that liquefy at these temperatures (Dian et al., 2007; Lida et al., 2002). According to Leissner et al. (1991), the proportional relationship between starting hard-stock concentration and its effect on the temperature at which the greatest variation in SFC occurs in the interesterified blends is indicative of the increased heat resistance produced by the increase in saturated fatty acids.

Small alterations in SFC between 15 and 25 °C characterize plastic fats (Leissner et al., 1991). The plasticity of the interesterified blends diminished with increasing FHSBO concentration. The interesterified 90:10 SO:FHSBO blend showed low SFC at all the temperatures considered, endowing this sample with low plasticity. According to Petrauskaitė et al. (1998), this can be explained by its low percentage of saturated fatty acids (24.32%), which should be greater than 25–30% in order to assure minimum plastic properties.

The alterations in the SFC profiles resulting from interesterification can be correlated with the melting point results. Increased FHSBO concentration produced smaller alterations in the SFC profiles of the interesterified blends as compared with the originals, in the same way that it reduced the variation among the melting points of the blends before and after the reaction.

Identification of applications for new fats is guided initially by correlating their physical properties with the properties of commercial fats for specific applications. The SFC profile, particularly, has important implications for the characteristics of margarines and shortenings at various temperatures, given that it is the main tool for specifying and selecting fats for food applications (Wassell & Young, 2007).

At low temperatures (4 at 10 °C), SFC gives an indication of the fat's spreadability at refrigeration temperatures, which should not exceed 32% to 10 °C (Lida & Ali, 1998; Wassell & Young, 2007). The interesterified 60:40 SO:FHSBO blend had well above 32% SFC at 10 °C, indicating that it does not offer satisfactory spreadability for use in refrigerated products. SFC between 20 and 22 °C is related to the product's stability and its resistance to oiling-out and should be above 10% (Lida & Ali, 1998; Wassell & Young, 2007). The interesterified 70:30 and 60:40 SO:FHSBO blends fall within this group of fats. The percentage of solid fat above 25 °C represents the hardness of a specific fat, while the values between 35 and 37 °C demonstrate the potential for releasing aromas during melting (Lida & Ali, 1998; Wassell & Young, 2007). Therefore, the interesterified 70:30 and 60:40 SO:FHSBO blends are regarded as suited mainly to endowing products with structure and the 80:20 and 90:10 SO:FHSBO blends, with lower SFC values at 1% to 37 °C, are regarded as offering good melting characteristics at body temperature (Karabulut et al., 2004).

Petrauskaitė et al. (1998), Karabulut et al. (2004), Karabulut and Turan (2006), Ghotra et al. (2002), Wassell and Young (2007), Grimaldi et al. (2000), Timms (2003a), Dijkstra (2007) and Lee et al. (2008) present SFC curves for various fats and shortenings for a variety of industrial purposes. When compared with the SFC profiles encountered in the literature, the interesterified 90:10 SO:FHSBO blend exhibits characteristics similar to those of frying shortenings. As the frying process deposits fat on the surface of the foods, frying shortenings should be free of solids at body temperature in order to avoid the sensation of graininess in the mouth (Ghotra et al., 2002). The 80:20 SO:FHSBO blend had a melt profile close to those of fatty bases for table margarine production and meets the SFC requirements to assure spreadability under refrigeration and complete melting at body temperature for margarines of this type (Karabulut & Turan, 2006).

The interesterified 70:30 SO:FHSBO blend exhibited a SFC profile and plasticity suitable for use in bakery and confectionery shortenings. According to Wassell and Young (2007), shortenings

for these purposes should create stable emulsions, but have good creaming properties, which can be obtained when SFC ranges from 15% to 20% at 20 °C, as is the case with this sample.

Meanwhile, the interesterified 60:40 SO:FHSBO blend has a SFC profile similar to most all-purpose shortenings and biscuit-filling fats, which should offer high SFC at room temperature in order to endow suitable crystalline structure and resist deformation (Ghotra et al., 2002; Woerfel, 1995). However, the SFC of this blend at 40 °C is 8.91%. In data from the relevant literature, similar SFC values are encountered at this temperature for fats for use in tropical regions and lower values for most shortenings and fats for this purpose. The possibility should thus be considered that waxy mouthfeel may be associated with direct use of this interesterified blend. According to Jeyarani and Reddy (2003), List et al. (1995) and Rousseau et al. (1996), in many cases, an interesterified fat should be diluted with small proportions of liquid oil in order to adjust its physical properties. One sure option to avoid this problem would thus be to add small amounts (1–5%) of a liquid oil, such as soybean oil, to this fraction (Dijkstra, 2007).

The interesterified 50:50 SO:FHSBO blend exhibited significantly high SFC between 35 and 40 °C (28.72–22.55%) and at 50 °C (10.91%), precluding its use at body temperature or even direct application as a fatty base for shortenings (Dijkstra, 2007; Grimaldi et al., 2000; Karabulut et al., 2004).

### 3.5. Isosolid diagrams

When oils and fats with differing compositions are blended, a number of behaviors may be observed: solid solutions (compatibility among the fats), monotectic behavior or eutectic behavior (Timms, 2003b). The isosolid diagrams are useful in studying compatibility among different fats. The isosolid lines represent the temperature at which the SFC of a fat or blend of fats is constant (Campos, 2005). Fig. 4 shows the isosolid diagrams of the original and interesterified blends.

The isosolid diagrams for the original blends indicate monotectic behavior, which prevails in mixed systems of fats with very different melting points (differences greater than 20 °C). Monotectic

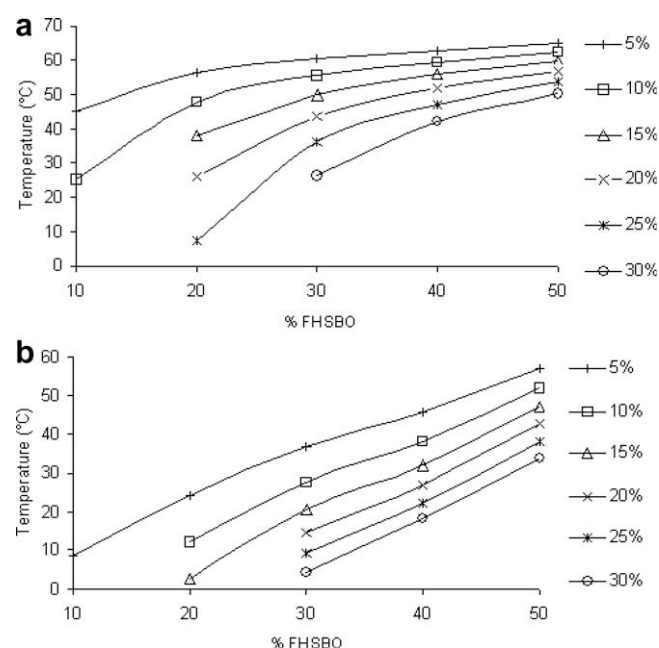


Fig. 4. Isosolid diagram of the blends before (a) and after (b) chemical interesterification.



behavior occurs when high melting point triacylglycerols are solubilized in the liquid triacylglycerol components (Rousseau & Marangoni, 1998; Timms, 2003b). In the original blends, FHSBO, with a melting point of 72 °C, is solubilized in the SO, whose melting point is around –22 °C (O'Brien, 2004). Monotectic behavior is associated with non-linear isosolid lines resulting from dilution of the solid fat by the liquid oil in a binary blend, as seen in Fig. 4a (Braipson-Danthine & Deroanne, 2006). This type of system was observed by Rousseau and Marangoni (2002) for lard/canola oil and palm oil/soybean oil blends and by Braipson-Danthine and Deroanne (2006) for canola oil/partly hydrogenated canola oil blends. When the temperature and the percentage of FHSBO in the blends increase, the isosolid lines converge to the right. The more closely spaced isosolid lines due to increased FHSBO concentration in the blends indicate that the effect of diluting is smaller when the trisaturated triacylglycerol content increases (Rousseau & Marangoni, 1998). This behavior was observed by Humphrey and Narine (2005) for a soybean oil/fully hydrogenated canola oil blend.

After interesterification, the substantial decrease in temperature recorded in the diagram indicates greater intersolubility in the solid state among the triacylglycerol species present, as a result of the greater heterogeneity in the triacylglycerol composition and increased proportion of triacylglycerols with intermediate melting points (Rousseau & Marangoni, 1998). Randomization resulted in linear evolution of the isosolid lines, which characterizes totally compatible interesterified blends and the formation of continuous solid solutions, as seen in Fig. 4b (Braipson-Danthine & Deroanne, 2003a).

No depressions were observed in the isosolid lines for the original and interesterified blends. Depressions in isosolid diagrams characterize the formation of a eutectic system, which results in liquefaction of the blend in specific compositions, representing problems in fat applications where consistency and SFC are important (Humphrey & Narine, 2005).

### 3.6. Consistency

The consistency of a semi-solid lipid system is a function of its SFC content and the microstructural properties associated with that content. Modification of SFC and crystalline network by interesterification affects the sensorial properties and technological performance of fats, thus directing their application in food products (Piska et al., 2006).

Table 4 shows the consistency of the blends with temperature, represented as yield value (gf/cm<sup>2</sup>), before and after interesterification. It was not feasible to ascertain the consistency of the 90:10

SO:FHSBO and 90:10 SO:FHSBO-I blends, because of these samples' low melting point.

Generally, interesterification led to a reduction in yield values for all the blends at all temperatures considered, with the sole exceptions of the 70:30 and 60:40 SO:FHSBO, where consistency increased at 5 °C. Consistency decreased with increasing temperature, which causes the gradual melting of the crystals and consequent destruction of the crystalline network which endows the fat with plasticity (Deman, 1983).

Consistency proved proportionally dependent on FHSBO concentration in the blends, before and after randomization. This behavior was also observed by Piska et al. (2006) in developing shortenings from various fully hydrogenated fats. According to Chiu and Gioielli (2002) and Larsson and Quinn (1994), the increase of saturated fatty acids content of a sample strongly influences its consistency, due to their high melting point.

The greatest alterations in yield values of the interesterified blends as compared with the original blends was observed at 15, 35, 50 and 55 °C for the 80:20, 70:30, 60:40 and 50:50 SO:FHSBO blends, respectively. At 25 °C, the original blends gave yield values ranging from 719.2 to 20535.7 gf/cm<sup>2</sup>; after interesterification, these values ranged from 0 to 8709.9 gf/cm<sup>2</sup>.

According to Haighton (1959), a fat is plastic and spreadable at yield values from 200 to 800 gf/cm<sup>2</sup>. The interesterified 80:20 SO:FHSBO blend, with a yield value of 309.6 gf/cm<sup>2</sup> at 10 °C, has satisfactory plasticity and spreadability properties for use at refrigeration temperatures, in addition to meeting SFC and melting point requisites for use in margarines, as mentioned earlier. Between 25 and 30 °C, the interesterified 70:30 SO:FHSBO blend was satisfactorily spreadable (yield values between 800 and 1000 gf/cm<sup>2</sup>), but exhibits ideal plasticity at 35 °C, which is important for sensorial properties such as mouthmelt sensation and lack of adhesiveness. Thus, its consistency profile from 25 to 35 °C corroborates its suitability for application in bakery and confectionery products. The interesterified 60:40 SO:FHSBO blend can be classified as hard at room temperature, with a yield value between 3352.4 and 2519.6 gf/cm<sup>2</sup> in the 25 to 35 °C interval; and is just above the spreadability limit (1500 gf/cm<sup>2</sup>) at 40 °C. According to Jeyarani and Reddy (2003), fats considered hard at room temperature are suitable for firmer food products, where deformations must not occur during handling or stocking. With a yield value below 1500 gf/cm<sup>2</sup> only from 45 °C up, the interesterified 50:50 SO:FHSBO blend can be considered too hard for direct application in foods (Haighton, 1959).

A number of recent studies have shown a direct relation between SFC and fat consistency (Rodrigues et al., 2003; Silva & Gioielli, 2006; Braipson-Danthine & Deroanne, 2006; Shi, Liang, &

**Table 4**  
Yield value (gf/cm<sup>2</sup>) of the SO: FHSO blends before and after interesterification.

T (°C)	Yield value (gf/cm <sup>2</sup> ) SO:FHSBO (%w/w)							
	80:20	80:20-I <sup>†</sup>	70:30	70:30-I	60:40	60:40-I	50:50	50:50-I
5	1891.3	1163.7	6829.5	7601.0	12647.1	17634.7	23789.8	21560.1
10	1668.9	309.6	5946.4	3120.1	11490.4	8174.5	23648.7	15552.4
15	1303.8	105.4	4791.2	1774.4	10904.2	7634.1	21527.9	14298.5
20	1170.4	–	4610.0	1263.6	10456.3	5476.3	20756.4	11737.4
25	719.2	–	4278.4	1069.1	10181.9	3352.4	20535.7	8709.9
30	601.7	–	3466.5	803.1	7473.6	2987.4	17248.8	6374.6
35	273.2	–	2234.4	273.4	5866.4	2519.6	13481.5	4402.7
40	–	–	2019.3	–	5222.1	1576.2	11735.7	2154.8
45	–	–	1606.9	–	4976.8	698.6	10709.8	1881.2
50	–	–	254.1	–	2591.4	280.4	5514.3	925.1
55	–	–	–	–	1413.3	–	2862.0	435.4
60	–	–	–	–	366.4	–	854.2	226.4

I<sup>†</sup>: Interesterified blend.



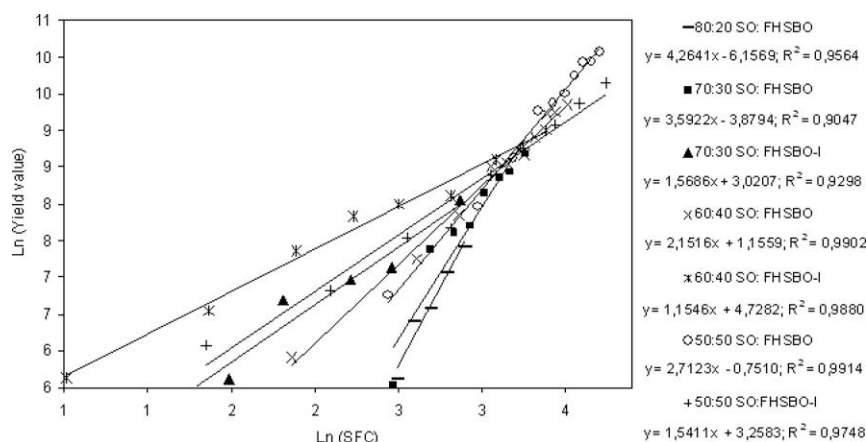


Fig. 5. Linear relations between the logarithm of yield value and the logarithm of SFC measurements for the original and interesterified blends.

Hartel, 2005; Gioielli, Simões, & Rodrigues, 2003; Braipson-Danthine & Deroanne, 2003b, 2004). Fig. 5 shows the relation between the logarithm of yield value and the logarithm of SFC for the samples at the temperatures at which consistency was evaluated, as proposed by Braipson-Danthine and Deroanne (2003b, 2004, 2006) for studying these properties. The relationship is not shown for the interesterified 80:20 SO:FHSBO blend, because the consistency of this sample was measurable only up to 15 °C, with 10 °C as the only temperature in common with SFC determination.

A significant ( $p < 0.05$ ) linear relationship was observed between the logarithm of consistency and the logarithm of SFC of the samples, over the temperature range considered, with a correlation coefficient ( $R^2$ ) greater than 0.9 in all cases. Braipson-Danthine and Deroanne (2003b, 2004, 2006) also observed this type of relationship for several binary and ternary blends of various oils and fats. They remark that significant linear relations between these magnitudes indicate that consistency is strongly dependent on SFC and can be used for predictive purposes.

#### 4. Conclusion

This study demonstrated that chemical interesterification significantly modified the triacylglycerol composition, and consequently the melting point, solid fat content and consistency properties of the SO:FHSBO blends. The interesterified blends displayed characteristics suited to a variety of industrial purposes, offering alternatives to partly hydrogenated fats. The interesterified 90:10, 80:20, 70:30 and 60:40 SO:FHSBO blends displayed characteristics suited to application as liquid shortening, table margarine, bakery/confectionery fat and all-purpose shortenings/biscuit-filling base, respectively.

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